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(19)

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(54) WALL COVERINGS

(71) We, MONTEDISON SPA, a Body Corporate organised and existing under the laws of Italy, of 31 Foro Buonaparte, Milan, Italy, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to wall coverings and a process for their manufacture with permanent embossing and high porosity. Porosity or transpirability are of importance in wall coverings.

The invention provides a process for the manufacture of a wall covering which comprises preparing a sheet from a mixture comprising up to 90% by weight of cellulose fibres and at least 10% by weight of fibrils of thermoplastic polymer, the fibrils having a surface area greater than 1 m^2/g, and subjecting the sheet to embossing with both the embossing device and the sheet at a temperature lower than the softening temperature of the thermoplastic polymer, and to heating at a temperature equal to or higher than the softening temperature of the thermoplastic polymer, the heating optionally being performed before the embossing.

The fibrils used may be homopolymers of monomers such as olefins (for instance low or high density polyethylene, polypropylene, or poly - 4 - methyl - 1 - pentene), acrylonitrile, vinylchloride or a vinyl monomer in general, an amide, an acrylic resin, a polyester resin, a polyurethane, polycarbonate, polyether, or a copolymer of two or more of such copolymerizable monomers.

The fibrils used may contain, incorporated therein an inorganic filler such as: kaolin, talcum powder, calcium sulphate, titanium dioxide and/or other inert material. The filler may be introduced into the fibril during formation. The quantity of inorganic filler in each fibril may be up to 70% by weight of the total weight of the fibril, the remaining 30% being thermoplastic polymer.

The cellulose fibres used may be derived totally from mechanical cellulose pulp or from chemical or semi-chemical cellulose pulp, or they may be derived from a mixture of two or more of these different types of cellulose. The weight ratio between the cellulose fibres: thermoplastic fibrils in the sheet may be from 90:10 to 10:90, but is preferably from 70:30 to 30:70.

The preparation of the sheet may be carried out according to the conventional techniques of the paper industry, starting from either an aqueous suspension or a suspension in another inert liquid medium, of a mixture of cellulose fibres and fibrils, using continuous or discontinuous machines. Preferably there are used aqueous suspensions containing from 0.7 to 1.5% by weight of total fibrous material, to which there may be added additives used in the conventional preparation of paper, for instance sizing agents, natural or synthetic, and inorganic fillers such as kaolin, talcum powder, or titanium dioxide, for example.

During its preparation, the sheet may be subjected to a "size press" operation in order to improve its printability and surface characteristics. On the other hand, a surface treatment may be carried out using a titanium dioxide suspension or a suspension of other pigments giving a high covering and opacifying power, at a concentration of from 10 to 50 g/l, in a solution of natural or synthetic binder. The surface treatment, similar to a coating operation, favours subsequent surface treatment, particularly printing, to which the sheet may possibly be subjected.

The process of the invention can be carried out by first subjecting the sheet to embossing and then to heating. When this method is used it is preferable, but not strictly necessary,

for the sheet to have, at the moment when it is subjected to the embossing, a water content of from 2% to 10%, preferably from 4% to 6%, calculated on the total weight of the sheet. This may be attained by passing the sheet through a drying oven maintained at a temperature lower than the softening temperature of the thermoplastic polymer from which the fibres are made.

Alternatively, the process can be carried out by first subjecting the sheet to the heating, then cooling the sheet to a temperature lower than the softening temperature of the thermoplastic polymer, and finally subjecting the sheet to the embossing.

Whatever method is used, the embossing is carried out at a temperature lower than the softening temperature of the thermoplastic polymer, or if the fibrils are of more than one thermoplastic polymer, at a temperature lower than the softening temperature of the thermoplastic polymer having the lowest softening temperature. Accordingly, the embossing can be carried out at room temperature, or below. The embossing may be preceded by printing, for example by rotogravure or flexography.

The embossing can be carried out by passing the sheet between two cylinders (rollers) of which one is an embossing roller which in general is made of steel, and the other cylinder is just a counter roller and may be made of hard rubber, for instance of neoprene, or of paper-wool, for example. The counter cylinder may, in its turn, be smooth or embossed with a relief or embossing complementary to the other cylinder. The pressure exerted on the sheet depends on the thickness and on the physical characteristics of the sheet itself. In most cases good results are achieved with operational pressures of from 10 to 100 kg/cm².

The heating causes the softening or the melting of the thermoplastic fibrils and leads to a very high porosity. The heating can be effected by passing the sheet through an oven, or under a set of infrared lamps, or even over the surface of a heated roller. The heating must be at least to the softening temperature of the polymer from which the fibrils are made, and preferably to that at which melting of the thermoplastic polymer occurs, or higher. Temperatures higher by at least 5°C, but preferably higher by from 20° to 40°C than the melting temperature of the thermoplastic polymer from which the fibrils are made are preferred.

If the sheet has been prepared from fibrils of different thermoplastic polymers, it is preferable to carry out the heating to a temperature at least equal to the softening temperature of the polymer having the highest softening point. The duration of the heating must be sufficient for softening or preferably melting at least a part of the fibrils incorporated in the sheet. It is sufficient for only the surface of the sheet to be brought up to a temperature at least equal to the softening temperature of the thermoplastic polymer.

After both embossing and heating, the sheet may be subjected to further decorating and/or printing processes, and may be provided on the side that will adhere to the wall with an adhesive.

The following Examples illustrate the invention (all % being by weight unless otherwise specified), and the properties of the sheets prepared are set out in the following Table.

EXAMPLE 1.

There was prepared a 1.5% aqueous suspension of a mixture of fibres consisting of: 50% of conifer cellulose pulp, and 50% of high density polyethylene fibrils having a melt index (M.I.)=5, a softening temperature of 118°C and a melting temperature=135°C. The fibrils contained incorporated in them 30% of kaolin. They had a length of from 1.4 to 1.6 mm, an apparent diameter (mean diameter) of from 15 to 25 micron, and a surface area of about 5 m²/g. The fibrils were prepared starting from a solution of the polyethylene in *n*-hexane, containing 30% of kaolin with a mean particle size of about 1.5 micron, by flash-spinning under the action of a high-speed inclined gas jet according to our British Patent Specification 1,392,667. The aqueous dispersion of fibres also contained 3% of a sodium resinate (glue) and 7% of homogeneously dispersed powdered kaolin.

Using a continuous paper machine, there was prepared from this dispersion a 150 g/m² sheet with a voluminosity of 1.95 cc/g. The sheet was left to dry at room temperature to a moisture content of about 6%. The sheet was then embossed by passing it continuously, at a constant speed, between an embossed steel cylinder and a resilient paper-wool cylinder having a 90° S.A. (Shore, scale A) hardness. The pressure exerted on the sheet was 50 Kg/cm². During the embossing operation, both the sheet and the two cylinders were kept at 20°C. The sheet thus obtained had an embossing which strictly reproduced in depth the pattern of the surface of the embossing cylinder. The embossed sheet was then conveyed to an oven heated at 160°C where it remained for 6 seconds. After this time, the sheet was removed from the oven and cooled down, wound on reels and transformed into coils for use.

EXAMPLE 2.

An aqueous dispersion at 1.5% concentration was prepared of a mixture of fibres consisting of: 20% coniferous cellulose fibres, 45% latifolia cellulosic fibres, and 35% high density polyethylene fibrils having a M.I.=20,

a softening temperature of 118°C and a melting temperature of 135°C.

The polyethylene fibrils did not contain any incorporated filler. They had a length of from 1.4 to 1.6 mm, an apparent (mean) diameter of from 15 to 25 micron and a surface area of about 5 m^2/g. These fibrils were prepared in the same way as in Example 1 but in the absence of kaolin. To the aqueous fibre dispersion there was admixed 3% of a sodium resinate and 10% by weight of powdered kaolin.

From this homogeneous dispersion, using a continuous paper machine, there was prepared a 150 g/m^2 sheet. This was treated on the same machine with "size-press", with an aqueous 2% solution of natural starches to improve the surface receptivity to ink. The sheet, whose voluminosity amounted to 1.5 cc/g, was subjected to printing on a conventional six-colour rotogravure printing machine, and embossed at 20°C while having about 10% moisture content, by passing between an embossing steel cylinder and a resilient neoprene cylinder having a 60° S.A. hardness at an operational pressure of 100 kg/cm^2. The embossed sheet was then passed into a hot air oven heated at 175°C, where it remained at that temperature for 5 seconds, after which it was cooled down and wound.

EXAMPLE 3.

By mixed beating up to 30° S.R. (Schopper-Riegler) there was prepared an aqueous 1% dispersion of fibres consisting of: 15% coniferous cellulose, 15% latifolia cellulose, and 70% polypropylene fibrils with an isotacticity index of 90%, M.I.=10, a softening temperature of 130°C and a melting temperature of 170°C.

These fibrils were produced as in Example 1. They contained 40% of incorporated kaolin. They had an average length of about 1.5 mm, apparent (mean) diameter of about 20 micron and a surface area of about 3.5 m^2/g. The aqueous fibre dispersion contained 3.2% of sodium resinate and 5% of kaolin dispersed therein.

Using a continuous flat-table machine having a width of 2.5 m at an operational speed of 150 m/min., from the above indicated dispersion there was prepared a sheet of 150 g/m^2. The sheet obtained had a voluminosity of 1.95 cc/g. The sheet was embossed at room temperature, by passing it over an embossing cylinder coupled to an opposing "paper-wool" roller. The pressure exerted on the sheet was 90 kg/cm^2. The resulting sheet was passed between plates heated by infrared rays so as to attain 200°C. It was kept at this temperature for about 5 seconds, and then again cooled down and wound up on a reel for final packaging.

EXAMPLE 4.

On a standard (conventional) paper machine, by mixed beating at 28° S.R., there was prepared an aqueous 1.5% dispersion of a fibre mixture: 25% conifer cellulose pulp, 25% latifolia cellulose pulp, 8% wood pulp, and 42% high density polyethylene fibrils having a M.I.=30, a melting temperature=135°C, and a softening temperature of 118°C.

The fibrils contained incorporated in them 30% of kaolin. They had a mean weight length of 1.6 mm, an apparent diameter (mean diameter) of 18 micron and a surface area of about 5 m^2/g.

These fibrils had been prepared starting from a solution of the polyethylene in *n*-hexane, containing 30% of kaolin with a mean particle size of about 1.5 micron, by flash-spinning under the action of an inclined high-speed gas jet as in Example 1. The aqueous fibre dispersion contained 2% of sodium resinate and 1% of Aquapel (Trade Mark) (adhesive). By means of a continuous, flat-table machine, 2.5 m wide, and at an operational speed of 150 m/minute, with the dispersion there was prepared a sheet with a weight of 150 g/m^2. The sheet thus obtained had a voluminosity of 1.80 cc/g.

This sheet was passed through a forced hot air oven at 50 m/min and at 140°C. The dwell period in the oven was 10 seconds. The sheet was cooled down to room temperature (25°C) and embossed by passing it between an embossing steel roller and a resilient paper-wool cylinder having a hardness of 90° S.A. at the same room temperature. The pressure exerted on the sheet was 50 kg/linear cm. The thus finished sheet was then wound onto coils and cut up.

EXAMPLE 5.

Following the same procedures in Example 1, there was prepared a sheet containing 55% of synthetic polypropylene fibrils (M.I.=20, softening temperature=122°C, melting temperature=168°C) having a weighted mean length of 1.8 mm, an apparent or mean diameter of 25 micron and a surface area of about 6 m^2/g. These synthetic fibrils contained incorporated in them 30% of kaolin having a mean particle size of about 1.5 micron. During the preparation stage on the flat plane machine, the sheet was treated in a size-press with an aqueous solution of starch containing in suspension 50 g/l of titanium dioxide to give the sheet good surface properties. The sheet obtained had a voluminosity of 1.9 cc/g.

The sheet was then passed through an infrared radiation device at 50 n/min. to bring the sheet up to 178°C. At the outlet of the infrared plate, the sheet was subjected to a

smoothing operation to improve its printability. The sheet was passed while the synthetic material was still in the thermoplastic phase, between two rollers of a calender, one of the rollers being of smooth sanded steel and cooled with water, while the other roller was made of rubber and had 65 S.A. hardness.

The sheet thus obtained had a printable surface, with a smoothness of 85 cc/min. (measured according to the ATICELCA (Associazione Technici Italiani Celluloa e Carta) MC 16 Standards); it was left to cool down and was then printed on a rotogravure six-colour machine and trimmed. The embossing operation was carried out continuously at the same speed as the printing speed (125 m/min) between two rollers, one of steel and carrying engraved the pattern to be reproduced, the other made of paper-wool and carrying the negative of the pattern to be embossed. The cylinders and the sheet were kept at 23°C. The pressure exerted on the sheet was about 50 Kg./linear cm.

TABLE

Characteristics	Measurement unit	Example 1	Example 2	Example 3	Example 4	Example 5
Weight	g/m^2	149.7	141.2	145	133.5	143.2
Thickness	microns	357	300	340	231	282
Longit. breaking load in dry condition	Kg	5.48	9.17	4.18	5.58	6.31
Trans. breaking load, dry condition	Kg	3.15	5.27	2.68	3.08	3.20
Longit. breaking load, wet condition	Kg	2.83	3.73	3.98	2.80	3.64
Trans. breaking load, wet condition	Kg	1.89	2.42	2.17	1.7	1.8
Residual longit. resistance	%	52	41	95	49	57.6
Residual trans. resistance	%	60	46	81	45	43.7
Longit. elongation	%	1.5	1.6	1.3	1.9	1.56
Trans. elongation	%	4.6	4.5	2.7	4.8	not determined
Permeability to water	$\frac{g.\,mm}{m^2\,24h}$	376	374	410	367	400
Permeability to steam	$\frac{g.\,mm}{m^2\,24h}$	175	113	196	158	202
Bendtsen porosity to air (measured according to ATICELCA MC 19 Standards)	cc/min.	941 ±41	700 ± 85	1100 ± 70	850 ± 41	950 ±45
Loss of embossing	cycles	70	30	700	50	100
Tearing in the wet	cycles	128	60	1000	90	300

WHAT WE CLAIM IS:—

1. Process for the manufacture of a wall covering which comprises preparing a sheet from a mixture comprising up to 90% by weight of cellulose fibres and at least 10% by weight of fibrils of thermoplastic polymer, the fibrils having a surface area greater than 1 m^2/g, and subjecting the sheet to embossing with both the embossing device and the sheet at a temperature lower than the softening temperature of the thermoplastic polymer, and to heating at a temperature equal to or higher than the softening temperature of the thermoplastic polymer, the heating optionally being performed before the embossing.

2. A process according to claim 1 in which the heating is at a temperature at least 5°C higher than the melting temperature of the thermoplastic polymer.

3. A process for the manufacture of a wall covering as herein described in any of the Examples.

4. A wall covering manufactured by a process according to any preceding claim.

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